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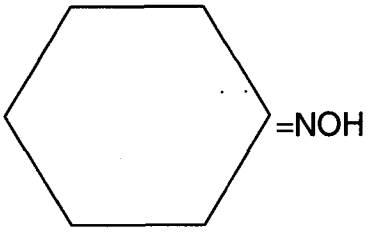

CYCLOHEXANONE OXIME

CAS NUMBER 100-64-1

USEPA HPV CHALLENGE PROGRAM SUBMISSION
(FIRST DRAFT)

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TEST PLAN

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EXECUTIVE OVERVIEW

Cyclohexanone oxime, a white crystalline solid, is used primarily as a captive intermediate in the synthesis of caprolactam which, in turn, is polymerized to polycaprolactam (Nylon-6) fibers, resins and plastics. Recent annual production figures for cyclohexanone oxime are not available.

Based on the fact that cyclohexanone oxime is a "closed-system intermediate," and because occupational exposure and releases to the environment are minimal, DSM Chemicals will be providing detailed information in this HPV Test Plan in support of a claim for "reduced testing requirements" for this oxime. This information can be found in an APPENDIX to this Test Plan (See pp. 18-30) entitled "Substantiation of Closed System Intermediate Status."

Adequate data for cyclohexanone oxime are available relative to Physical/Chemical properties. This oxime will be a solid below its melting point (190-196°F) and a liquid above this point. Based on its low vapor pressure (0.029 mm Hg), high boiling point (406°F), and aqueous solubility (1.5 wt%), it will tend to remain in water and only slowly volatilize.

Relative to Environmental Fate and Pathways, limited data are available. Cyclohexanone oxime is stable in water and will hydrolyze only at sustained temperatures (250-300°F). Some data exists on photo-oxidation but there is no information on biodegradation or transport and distribution between environmental compartments. Relative to the category of Ecotoxity, valid date exists for the fathead minnow (96-hr LC50=208 mg/L) but no toxicity information was found for invertebrates and algae. Although most of the preceding studies are not adequate to meet SIDS/HPV requirements, no testing is recommended for the categories of "Environmental Fate and Pathways" and "Ecotoxicity" because cyclohexanone oxime is a "closed system intermediate" and poses no major hazard relative to releases into the environment.

Acute toxicity to mammals appears to be low-to-moderate as demonstrated by an oral lethal dose (LD) in rats of >500mg/kg and a dermal

absorption LD50 in rabbits of >5000 mg/kg. On a repeated exposure basis, several subacute (2-week) and 90-day oral toxicity studies have been conducted in both rats and mice. In the preceding studies, the major target organs appear to be the erythrocyte, the spleen, the bone marrow and liver. Toxicokinetic studies by various routes of administration in rats suggest that cyclohexanone oxime is readily absorbed, subsequently metabolized, and then is excreted in the urine as glucuronides within a day. Relative to genetic toxicity potential, cyclohexanone oxime has been thoroughly tested in both *in vitro* and *in vivo* studies. The overall weight of evidence suggests that cyclohexanone oxime poses no genotoxic hazard. Relative to the HPV Program, adequate studies are available in the areas of "Acute Toxicity", "Repeated Dose Toxicity", and "Genetic Toxicity" and no additional testing is needed.

No definitive studies to assess the potential effects of cyclohexanone oxime on pregnancy or on the reproductive performance of male and female animals have been conducted. However, a determination by EPA that cyclohexanone oxime is a "closed system intermediate" with low occupational exposure potential will eliminate the need for any additional reproductive toxicity testing. A developmental toxicity study, on the other hand, will have to be conducted to fulfill HPV requirements for the "Reproductive/Developmental Toxicity" category. Such a study will be conducted in rats by the oral route and appropriate OECD guidelines will be followed.

Overall, cyclohexanone oxime as a "closed system intermediate" chemical does not appear to represent an unacceptable risk to human health or the environment. Under the EPA HPV Challenge Program, cyclohexanone oxime was evaluated, data gaps were identified, and a decision was made to conduct additional testing only in the area of "Developmental Toxicity". An appropriate study to meet the HPV requirement will reference OECD Guidelines and will be conducted starting in the 3rd or 4th quarter of 2006 and take less than a year to complete.

TESTING PLAN AND RATIONALE

Testing Plan in Tabular Format

Cyclohexanol Oxime	Information Available?	OECD Study?	GLP Study?	Other Study?	Estimation Method?	Acceptable?	Testing Recommended?	Comments
HPV Endpoint								
Physical/Chemical Properties								
Melting Point	Y	N	N	N	N	Y	N	
Boiling Point	Y	N	N	N	N	Y	N	
Vapor Pressure	Y	N	N	N	N	Y	N	
Partition Coefficient	Y	N	N	N	Y	Y	N	
Water Solubility	Y	N	N	N	N	Y	N	
Environmental Fate								
Photodegradation	Y	N	N	N	Y	N	N	*
Water Stability	Y	N	N	N		N	N	*
Transport	N						N	*
Biodegradation	N						N	*
Ecotoxicity								
96-Hour Fish	Y	N	N	N	N	Y	N	*
48-Hour Invertebrate	N						N	*
72-Hour Algae	N						N	*
MammalianToxicity								
Acute Toxicity	Y	Y/N	Y/N	Y	N	Y	N	
Repeated Dose	Y	Y?	Y		N	Y	N	
Genotoxicity (Point Mutation)	Y	Y?	Y	N	N	Y	N	
Genotoxicity (Chromosome Aberration)	Y	Y	Y	N	N	Y	N	
Reproductive Toxicity	Y	N	N		N	N	N	*
Developmental Toxicity	N						Y	Oral rat; OECD Protocol

*Based on claim of "closed system intermediate" status of cyclohexanone oxime and very low potential for both occupational exposure and environmental releases. See attached APPENDIX (Starting on p. 18).

INTRODUCTION

Cyclohexanone oxime, CAS No. 100-64-1, is a chemical intermediate used primarily in a closed system in the production of caprolactam. The latter chemical is subsequently polymerized to produce Nylon-6 (polycaprolactam) fibers, resins, and plastics.

As part of this HPV Test Plan, DSM Chemicals North America, a primary producer and the HPV Sponsor of cyclohexanone oxime, has provided detailed information in support of a claim for reduced testing requirements for this "closed system intermediate". This information is contained in an APPENDIX to this Test Plan (See pp. 18-30) entitled: "Substantiation of Closed System Intermediate Status." Acceptance of such a status will result in a reduced SIDS testing plan for cyclohexanone oxime.

Various studies have already been conducted on the toxicity of cyclohexanone oxime. Those studies (key and other supporting studies) are summarized in this document with comments as to whether or not they meet the requirements of the USEPA High Production Volume (HPV) Program. Robust summaries, using a SIDS format, have been prepared and include detailed information on key studies and some supporting studies; these detailed summaries are contained in a separate document (Tier 1 Screening SIDS DOSSIER on the HPV Phase....Chemical).

PHYSICAL-CHEMICAL DATA

Physical/chemical properties for cyclohexanone oxime are available from the literature and from the manufacturer:

Melting Point	190-196°F (1)
Boiling Point	406°F (1)
Vapor Pressure	0.029 mm Hg @ 77°F(1)
Partition Coefficient	Log P_{ow} = 0.84 @ 77°F(2)
Water Solubility	1.5 wt% @ 68°F(1)

Cyclohexanone oxime (MW=113.18) is a 6-carbon ring with an "NOH" group on C1. It is characterized as a white solid at room temperature and as a clear-to-white crystalline liquid above its melting point of 190-196° F(1). It has a specific gravity (water=1) of 0.97 and a pungent-to-slightly sweet odor (1). Cyclohexanone oxime also has a calculated Henry's Law Constant of 8.05E-06 atm-m^3/mole (@ 25°C)(2). It also has a lower flammability limit of 1.3%, a flash point (closed cup) of 181.4°F and autoflammability temperature of 545° F (1).

Recommendation:

No additional studies are recommended to fulfill the HPV required end points for "Physical/Chemical Properties".

ENVIRONMENTAL FATE AND PATHWAYS

Atmospheric photo-oxidation may be an important removal process for cyclohexanone oxime. It has a calculated atmospheric OH constant of 7.07E-12 cm^3/molecule-sec (2). Relative to stability in water, a manufacturer's MSDS states that the chemical is stable and that hydrolysis occurs only at sustained temperatures (250-300°F)(1). No information was available on Transport and Distribution between Environmental Compartments and no information was available on biodegradation.

Recommendation:

Although none of the proceeding studies are adequate to meet SIDS/HPV requirements, no additional testing is recommended since cyclohexanone oxime is a "closed system intermediate" and poses no major concerns relative to releases into the environment.

ECOTOXICITY

Acute aquatic toxicity data are available for cyclohexanone oxime in fish. In a study following flo-through guidelines, the 96-hr LC50 based on survival for the fathead minnow (Pimephales promelas) was 208 mg/L (3). No information was available on invertebrates or algae.

Recommendation:

Although the preceding fish toxicity data may meet HPV requirements, there is no information on invertebrates or algae. However, no additional testing is recommended since cyclohexanone oxime is a "closed system intermediate" and poses no major concern relative to environmental releases.

MAMMALIAN TOXICITY

A. Acute Toxicity

The acute toxicity potential of cyclohexanone oxime has been evaluated by several routes of administration. By the intraperitoneal route, its LD50 in mice was 250 mg/kg (3). By an unspecified route of administration, an LD50 of 710 mg/kg was reported for male mice (4).

When cyclohexanone oxime was given orally to rats, its "LD" was reported as >500 mg/kg (5). This value for oral toxicity is supported by results from a 10-dose subacute oral study at 300 mg/kg showing no mortality in rats (6).

By the dermal route of administration, the dermal absorption LD50 in rabbits was >5000 mg/kg, the highest dose tested. Although rabbits showed no adverse clinical signs or body weight changes, various red blood cell parameters were affected and methemoglobin was elevated at all dose levels (800, 2000 and 5000 mg/kg), suggesting that cyclohexanone oxime may be absorbed through the skin in toxicologically significant amounts (7).

There were no reliable data found on inhalation toxicity potential. However, based on the "closed-system intermediate" status of cyclohexanone oxime, inhalation exposure of workers does not present a significant hazard.

Recommendation:

The preceding acute toxicity studies by the oral, dermal and intraperitoneal routes are adequate to fulfill HPV requirements for "Acute Toxicity".

B. Repeated Dose Toxicity

Several repeated dose toxicity studies on cyclohexanone oxime have been conducted by the oral route by both gavage and drinking water administration.

Two 2-week gavage studies were conducted in rats showed dose-related erythroid hyperphasia in the spleen and bone marrow. In one study (8), Sprague-Dawley rats that received 1, 10, or 1000 mg cyclohexanone oxime per kg body weight for 2 weeks had hematologic differences including lower erythrocyte counts, higher platelet counts, lower hemoglobin concentrations and hematocrit levels, and greater mean red cell hemoglobin and mean red cell volume values than the control values. Bone marrow smears indicated lower myeloid, lymphocyte, and monocyte counts concomitant with elevated erythroid counts. There was also general splenic enlargement with hematopoietic cell proliferation.

In a second study (6), male and female F344 rats that received 10, 25, 75, 150, or 300 mg cyclohexanone oxime per kilogram body weight by gavage for 2 weeks had adverse hematologic changes similar to those of the Sprague-Dawley rats. Observations included a dose-related decrease in erythrocyte counts with concomitant increases in the numbers of circulating nucleated erythrocytes and reticulocytes and reduced hematocrit levels and hemoglobin concentrations. Methemoglobin concentrations, measured at the highest dose, were significantly elevated. The rats were observed for another 2 weeks without compound

administration. By Day 28, hematologic values in females had returned to normal and males displayed only slightly depressed erythrocyte counts and mildly elevated reticulocyte counts. No significant effects on body weights and no clinical signs of toxicity were noted in males or females. Splenomegaly and hepatomegaly were observed in male and female rats on Day 14 and Day 28. The hematology results suggested that the hematotoxic effects of cyclohexanone oxime administration were reversible following cessation of exposure. The authors theorized that cyclohexanone oxime induces oxidative damage to the erythrocyte resulting in hemolytic anemia compensated by increased erythropoiesis.

The results of 13-week oral toxicity studies in rats and mice were similar to those of the two-week oral studies with evidence of splenomegaly and erythroid hyperplasia in the spleen and bone marrow. In an oral gavage study (7), Fischer 344 rats (20/sex/dose) received doses of 0, 0.25, 2.5, and 25 mg cyclohexanone oxime per kilogram body weight five times a week for 13 weeks. All males survived to the end of the study; three of 20 females in the 25 mg/kg group died before the end of the study. Males were observed with clinical signs of toxicity that included persistent red nasal discharge (at 25 mg/kg only), chromodacryorrhea and swollen conjunctiva (at 2.5 and 25 mg/kg), and corneal opacity (at all dose levels). No significant effects on body weight or feed consumption were observed in males or females. Hematologic changes similar to those seen in the 2-week study were noted. Dose-related anisocytosis, poikilocytosis, elevated osmotic red blood cell fragility, and a greater incidence of Howell-Jolly bodies were observed. Splenomegaly was noted at necropsy, and histopathologic examination showed erythroid hyperplasia in the bone marrow and spleen and increased hemosiderin pigment deposition in the spleen. Data from satellite groups terminated at 30 and 60 days showed a NOEL at the lowest dose, but results from the end of the study showed a clear cumulative dose-response down to the 0.25 mg/kg dose level.

In a second 13-week toxicity study (9), B6C3F1 mice (10/sex/dose) were given drinking water containing 0, 625, 1,250, 2,500, 5,000 or 10,000 ppm

cyclohexanone oxime. Deaths occurred in the 10,000 ppm groups and weight gain was depressed in males and females given 10,000 ppm and in females given 5,000 ppm. There were significant increases in relative spleen weight at exposure levels of 5,000 and 10,000 ppm and significant increases in the relative liver weights of males and females that received 10,000 ppm. Microscopically, hemtopoietic cell proliferation was observed in the spleen of males and females in the 5,000 and 10,000 ppm groups. Centrilobular cell hypertrophy was observed in the liver of males in the 2,500, 5,000, and 10,000 ppm groups and in females in the 5,000 and 10,000 ppm groups. Olfactory epithelial degeneration was observed in all exposed groups. In summary, the major targets of cyclohexanone oxime were the erythrocyte, spleen, liver and nasal epithelium. The NOEL for erythrotoxicity is 2,500 ppm following 13 weeks of exposure. The NOEL for hematopoietic cell proliferation in the spleen is 2,500 ppm. The NOEL for hepatotoxicity is 1,250 ppm for males and 2,500 ppm for females following 13 weeks of exposure. Some nasal olfactory epithelial degeneration was observed at all exposure levels; only at 625 ppm in males was the incidence of this lesion not significantly different from that in the controls.

Recommendations:

The subacute and subchronic oral toxicity data on cyclohexanone oxime are adequate to meet the HPV requirements for "Repeated Dose Toxicity".

C. Genotoxicity

Negative results were obtained in earlier *in vitro* mutogenicity tests with several strains of *Salmonella tyhimurium*, with and without metabolic activation (10, 11) and with *Escherichia coli* strain WP2 (10). In a later point mutation assay (9), cyclohexanone oxime was mutagenic in *Salmonella typhimurium* TA1535 with hamster S9 activation but negative in the same strain with rat liver S9 and negative without any S9 activation. No evidence of mutagenicity was seen in strains TA97, TA98, or TA100 with or without rat or hamster S9

activation. Under similar experimental conditions (12) the same positive result in strain TA1537 was reproduced using hamster liver S9; similarly, no evidence of mutagenicity was seen in strain TA100 with or without hamster liver S9 activation.

In a non-bacterial, *in vitro* mutagenicity assay (9), cyclohexanone oxime tested negative for induction of chromosome aberrations with S9 activation and equivocal in the absence of rat liver S9. In one other *in vitro* assay (11), this oxime was positive in L5178Y mouse lymphoma cells without metabolic activation; the addition of rat liver S9 eliminated the mutagenic effect.

Relative to *in vivo* mutagenicity, cyclohexanone oxime was negative in an intraperitoneal mouse micronucleus study at doses (3 doses at 24 hour intervals) as high as 1000 mg/kg. In addition, this oxime was also negative in a micronucleus assay conducted on mice that were given the chemical at drinking water doses as high as 10,000 ppm for 90 days (9). In one other in vivo study (13), there was no increase in the frequency of sex-linked recessive lethal mutations in germ cells of male *Drosophila melanogaster* administered cyclohexanone oxime by feeding.

Based on an overall weight-of-evidence approach, cyclohexanone oxime is not mutagen.

Recommendation:

No additional testing is required. The HPV requirement for genetic testing has been fulfilled by the preceding *in vitro* and *in vivo* studies sensitive to both point mutations and chromosome aberrations.

D. Reproductive Toxicity

No definitive studies to assess reproductive performance of male and female experimental animals have been conducted on cyclohexanone oxime.

In a 90-day drinking water study (9) on cyclohexanone oxime, mice receiving drinking water containing as much as 5,000 ppm were given sperm

motility and vaginal cytology evaluations. There were no differences between treated and control mice. In addition, there were no histophathological effects seen in the reproductive organs of the male or female mice.

No other information on the reproductive toxicity potential of cylohexanone oxime was available.

Recommendation:

Although the preceding information does not meet the HPV requirements for "Reproductive Toxicity", no additional testing is recommended. If EPA accepts DSM North America's request to categorize cyclohexanone oxime as a "closed system intermediate", and the supporting data indicating very low exposure potential for both man and the environment, then reproductive toxicity testing will not be a requirement for this oxime.

E. Developmental Toxicity

No information on the developmental toxicity potential of cyclohexanone oxime was found in the toxicological literature (published or unpublished).

Recommendation:

Since a "closed-system intermediate" categorization of cyclohexanone oxime does not eliminate the HPV requirement for an adequate developmental toxicity study, DSM Chemicals North America will conduct such a study in rats, by the oral route, using the appropriate OECD guidelines.

F. Toxicokinetics

A toxicokinetic study (14) of cylochexanone oxime has been conducted in male Fischer 344 rats by three different routes of administration. The chemical was found to be rapidly absorbed and cleared within 24 hours after a single oral administration of 1, 10, or 30 mg/kg of [^{14} C]-cyclohexanone oxime in aqueous

solution. The majority of the cyclohexanone oxime-derived radioactivity was excreted in the urine. Three urinary metabolites were identified: cyclohexylglucuronide and the monoglucuronides of *cis*- and *trans*-cyclohexane-1,2-diol. Low levels of radioactivity (2%-3% of the dose) were retained in the tissues 24 hours after exposure. After intravenous administration of 1 mg/kg of [^{14} C]-cyclohexanone oxime, the oxime was rapidly cleared from plasma, with half lives of 1.6 minutes (alpha phase) and 18.2 minutes (beta phase). When cyclohexanone oxime was applied dermally (30 mg/kg), only 4% to 5% of the dose was recovered in the urine, feces, and tissues. The majority of the dose volatilized from the skin surface. However, the absorbed radioactivity was readily distributed and excreted, and its metabolic fate was no different than that observed after oral administration.

After a 14-day gavage study (8), cyclohexanone oxime has also been reported to induce increased microsomal activity (aniline hydroxylase and aminopyrine demethylase) in rats treated at a dose of 100 mg/kg body weight. In addition, cyclohexanone oxime has been reported to inhibit the oxidative metabolism of ethanol in rats and mice, an effect similar to that produced in humans as a result of disulfiram administration (15, 16, 17).

From the preceding animal studies, it is evident that cyclohexanone oxime can be absorbed by three different routes of administration. Most absorbed cyclohexanol is metabolized and is subsequently excreted as glucuronides.

CONCLUSIONS

Under the EPA HPV Challenge Program, adequate data to meet HPV requirements are available for cyclohexanone oxime relative to Physical/Chemical Properties, Acute Toxicity, Repeated Dose Toxicity, and Genotoxicity. Although the data available for Ecotoxicity and Environmental Fate and Pathways are limited, no additional studies in these areas are recommended since cyclohexanone oxime is a "closed system intermediate" and poses no exposure hazard relative to releases into the environment. The latter claim also

negates the need for the Reproductive Toxicity requirement. However, the "closed-system intermediate" status and low exposure potential does not alleviate the need for an adequate developmental toxicity study. Such a study, following OECD guidelines, will be conducted in rats by the oral route on cyclohexanone oxime.

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APPENDIX

SUBSTANTIATION OF CLOSED SYSTEM INTERMEDIATE STATUS FOR CYCLOHEXANONE OXIME

DSM Chemicals North America, Inc., hereby submits a claim for reduced SIDS testing needs for cyclohexanone oxime, a "closed-system intermediate." To support such a claim for reduced testing, the Company has provided detailed information on number of manufacturing sites, process descriptions, monitoring data, presence in products, and transport (if applicable) in this APPENDIX to the HPV Test Plan.

The format of this appendix consists of responses (along with diagrams and tabular data) to a required list of questions (excerpted from the SIDS manual). Based on these responses reflecting a very low-to-negligible exposure potential to workers and the environment, DSM Chemicals believes that the information requirements supporting an exemption claim for reduced SIDS testing have been satisfied. The information requirements follow on pages 19-30 of this document.

Information Requirements Supporting Exemption Claims for Reduced SIDS Testing Based on Exposure Considerations

I. Information on sites

A. Number of sites: **There is only one (1) site - DSM Chemicals North America, Inc. (DCNA) in Augusta, GA**

B. Basis for "closed process" conclusion at each site:

1) process description in enough detail to clarify the basis for claiming that the process is closed;

See Attachment 1 (p. 22-23) for a process description of cyclohexanone-oxime. A simplified block flow diagram of the process is provided in Figures 1 (p.24) and 2 (p.25).

2) if available, monitoring data showing no detection in any media, including the limits of detection;

As shown in Figure 1 (p.24), a small portion of cyclohexanone-oxime does come into contact with process water, which is discharged to our wastewater treatment plant (WWTP). Attachment 2 (p.26) is provided to show the cyclohexanone-oxime concentration in the combined feed to our on-site WWTP, including the monthly average and mean detection limit (MDL). Attachment 3 (p.27) shows the analysis for cyclohexanone-oxime in the WWTP effluent (Weir III). The analysis shows cyclohexanone-oxime at non-detectable (ND) levels, and a limit of detection is provided also.

3) if monitoring data are unavailable, a statement that no monitoring has taken place and the basis for believing, in the absence of data, that the chemical has not been released and that exposure does not occur.

Monitoring data for vapor emissions is unavailable. However, based on the low vapor pressure of

cyclohexanone-oxime (approximately 25 mmHg at normal operating temperature), and the fact that the surge vessel containing this product is heat traced and insulated, controlled at a fairly constant level, and equipped with a conservation vent, emissions of cyclohexanone-oxime are expected to be at de minimus levels. Tank emission calculation spreadsheets are provided in Table 1 (pp. 28-30) showing working losses to the atmosphere from each surge vessel below 0.4 lb/day.

C. Data on "presence in distributed product" or, in the absence of data, the basis for believing it is not present *at levels above trace concentrations.*

Cyclohexanone-oxime is used as an intermediate by DCNA to manufacture caprolactam. Attachment 4 (p.31) shows cyclohexanone-oxime analysis performed on our final product (caprolactam) storage tank year-to-date, including the yearly average, mean detection limit (MDL), and the internal DCNA Lab procedure.

II. Information on transport

If transport also occurs, then in addition to the above, the following should be provided :

- Mode of transport (e.g. water, truck, rail, pipeline)
- Volume (annual)
- Types of consignments (e.g. bulk or drums)
- Controls during transport and transfer at dispatching and receiving sites (placards, labels, etc.)

Not Applicable

III. Supporting evidence from a data search that the chemical is not present in other end products

To the best of our knowledge, cyclohexanone-oxime is used as an intermediate chemical in the manufacture of caprolactam. The caprolactam manufacturing process at DCNA is similar to that of our competitors, and as such, we are reasonably confident that their final product caprolactam will have similar analytical results showing only trace amounts of cyclohexanone-oxime in the final product as does DCNA (see Attachment #4 on p. 31).

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ATTACHMENT 1

Cyclohexanone-oxime, henceforth referred to as oxime, is an intermediate product formed in the production of caprolactam. Oxime is produced within the 2 HPO sections (Sections 26 & 36) of DCNA by the oximation of hydroxylamine (hyam) and cyclohexanaone (anone). The hyam is produced by the catalytic reduction of nitrate within the hyam reactor. Because hyam is unstable in a pure state, an aqueous solution of phosphoric acid, ammonium phosphate, and ammonium nitrate (referred to as Inorganic Process Liquor, or IPL) is used as its carrier. The anone is produced within the 2 Oxanone sections (Sections 35 & 45) of DCNA by the air oxidation of cyclohexane.

The oximation reaction takes place in 5 mixer-settler reactors where the hyam rich IPL stream is contacted with an organic stream of toluene and anone. The oxime produced in the reaction goes to the organic phase which leaves oximation with an approximate composition of 73% toluene, 25% oxime and 2% anone. This organic stream is washed with water and then distilled within two vacuum distillation columns. The oxime product (see Figure 1), recovered as the bottoms of the section distillation column, is then transferred to rearrangement where it is completely reacted, using oleum as a catalyst, to form caprolactam (see Figure 2). There are 2 rearrangement caprolactam purification sections (Section 27 & 37) at DCNA that further remove impurities and purify the caprolactam to a strength of ~100%.

The only accumulation points for purified oxime within the caprolactam production facility are the pumping vessels between distillation and rearrangement. These vessels, not capable of holding more than 5% of the respective plant's daily production capacity are used to provide just enough surge capacity to enable the safe shutdown of rearrangement or toluene-oxime distillation in the event of a process upset in either of the two sections. During normal operation, the level is controlled at a constant volume in the pumping vessel by making adjustments in the rearrangement section.

Points of release of oxime during the production of caprolactam include wastewater from the HPO sections and some vapor emissions, both of which are minimal. The presence of

oxime in the wastewater is primarily the result of the wash step of the toluene oxime and vacuum jet condensate from the toluene/oxime distillation. Prior to discharge, most of the oxime is removed from the washwater via a toluene extraction step. All of the wastewater is routed through a steam stripper, which also removes some oxime. This wastewater is subsequently treated within the site's biological wastewater treatment plant which removes residual oxime to below detectable limits in the plants effluent. The vapor release is limited to that coming from the oxime pump vessel which is limited because the vessel is controlled at a fairly constant level and is equipped with a conservation vent.

FIGURE 1

Rearrangement / Purification (27/37)

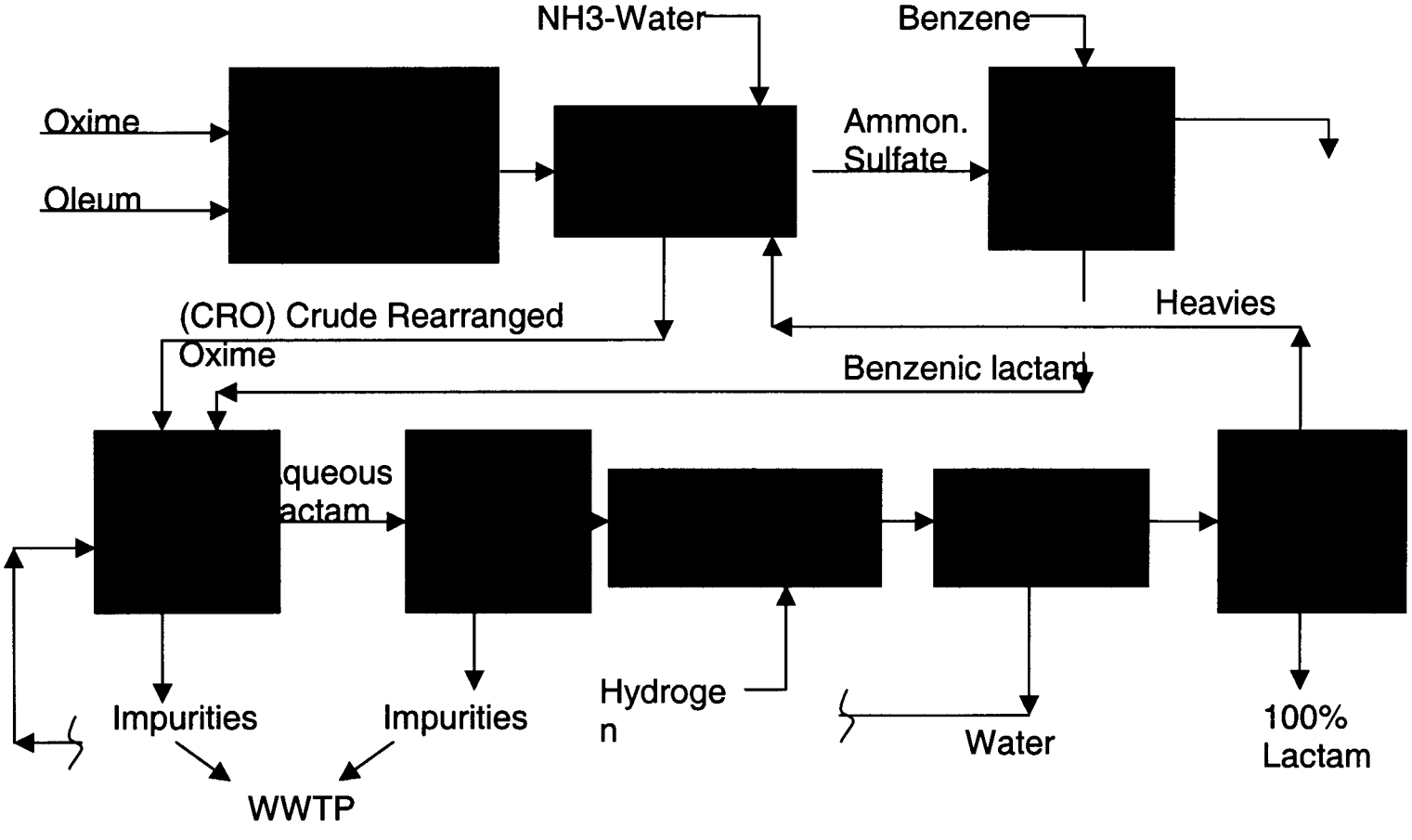
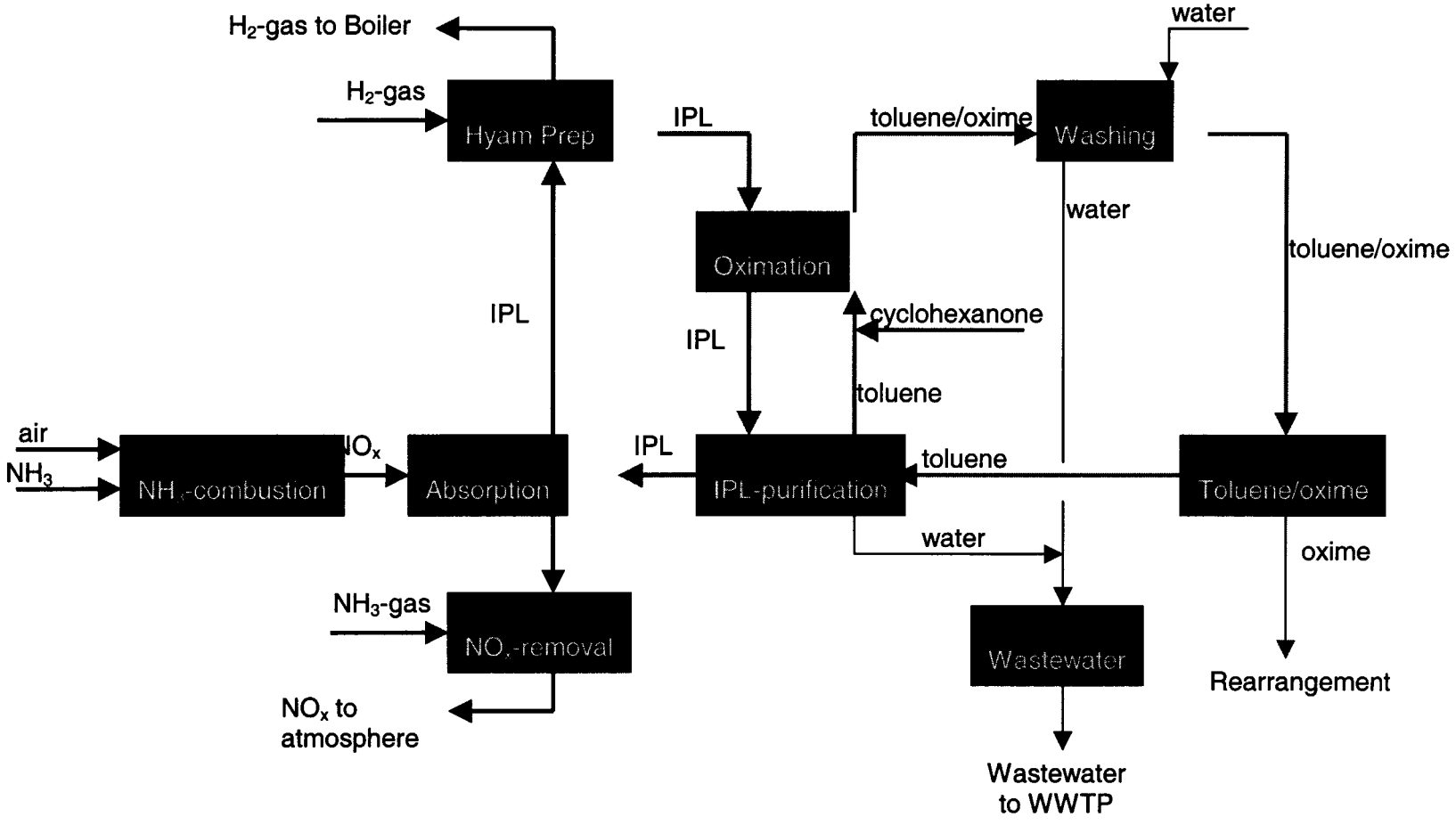


FIGURE 2

Hydroxylamine Phosphate Cyclohexanone Oxime (26/36)



1

ATTACHMENT 2

Combined Feed WWTP	
Date	**Oxime wt%**
10/31/05	0.0065
11/01/05	0.0081
11/02/05	0.0082
11/03/05	0.0080
11/04/05	0.0090
11/05/05	0.0061
11/06/05	0.0063
11/07/05	0.0096
11/08/05	0.0070
11/09/05	0.0105
11/10/05	0.0058
11/11/05	0.0047
11/12/05	0.0108
11/13/05	0.0097
11/14/05	0.0078
11/15/05	0.0094
11/16/05	0.0062
11/17/05	0.0047
11/18/05	0.0083
11/19/05	0.0101
11/20/05	0.0113
11/21/05	0.0069
11/22/05	0.0068
11/23/05	0.0070
11/24/05	0.0081
11/25/05	0.0074
11/26/05	0.0078
11/27/05	0.0072
11/28/05	0.0066
11/29/05	0.0050
11/30/05	0.0048
Average	**0.0076**
MDL	**0.0006**

Method
DCNA-10-GC047

ATTACHMENT 3

DSM Chemicals North America, Inc.

DSM Laboratory Special Analysis Request Report

DSMLAB: 9823

SUBMITTED: 11/22/2005 REPORTED: 11/29/200

ORIGINATOR: M. Ray

SAMPLE: Weir III

ANALYSIS: Cyclohexanone oxime

PURPOSE:

PRIORITY:

ANALYST: E. Moe APPROVED: Erin R. Moe

DISTRIBUTION: M. Ray, D. Morris, D. Smith, G. Bowen

cyclohexanone oxime; ppm ND

(limit of detection; 6 ppm)

TABLE 1 EXPLANATIONS

From: Pocta, John
Sent: Wednesday, December 28, 2005 8:57 AM
To: Morris, Dean
Subject: Oxime losses from V-2608/V-3608
Dean,

The oxime vapor emissions from oxime pump vessels V-2608/V-3608 are minimal for the following reasons:

1. Oxime has a low vapor pressure (approximately 25 mmHg at normal operating temperature),
2. The vessels are traced and insulated,
3. The vessels are equipped with conservation vents,
4. The vessels are controlled at a fairly constant level

Using the subsequent tables on pp. 28 & 29 (Tank Emission Calculation Forms), the estimated oxime emissions are less than 150 lb/yr from each vessel.

John

TANK NO. V-2608

Table 1
TANK EMISSION CALCULATION FORM

Tank No.	**V-2608**	**Tank type**	**Horizontal fixed roof (insulat**	**Date**	**03/04/06**
Material stored	**Oxime**	**Company**	**DSM Chemicals**	**Performed by**	
City	**Augusta**	**State**	**GA**		
Description	**Outdoor storage tank**				

INPUT DATA	Symbol		Units
Vapor pressure Antoine constants			
Constant A		**9.0490**	
Constant B		**2,992.500**	
Constant C		**273.150**	
Molecular weight	Mv	**113.2**	Lb/lb-mole
Tank design data			
Shell height	Hs	**9.00**	ft
Diameter	D	**8.00**	ft
Liquid height		9.00	ft
Avg. Liquid height	HL	**4.50**	ft
Tank volume		3,384	gallons
Turnovers	N	**41**	
Net throughput	Q	138,672	gallons/yr
Turnover factor	KN	0.899	
Working loss product factor	Kp	**1.00**	
Meteorological data			
Daily ave. ambient temp.	TAA	**N/A**	°F
Daily max. ambient temp.	TAX	N/A	°F
Daily min. ambient temp.	TAN	N/A	°F
Daily ambient temp. range	DTA	**N/A**	°F
Tank paint solar absorptance	α	**N/A**	
Daily total insolation factor	I	**N/A**	Btu/ft2-day
Liquid bulk temperature	TB	240.00	°F
Daily vapor temp. range	DTv	10.00	°F
Daily ave. liquid surface temp.	TLA	240.00	°F
Daily max. liquid surface temp.	TLX	242.50	°F
Daily min. liquid surface temp.	TLN	237.50	°F
VP @ daily ave. liquid surf. temp.	PvA	22.4063	mm Hg
VP @ daily max. liquid surf. temp.	PvX	23.8661	mm Hg
VP @ daily min. liquid surf. temp.	PvN	21.0264	mm Hg
VP @ daily ave. ambient temp.	Pamb	N/A	mm Hg
Daily vapor pressure range	DPv	2.84	mm Hg
Breather vent pressure setting range		**0.46**	psia
Breather vent pressure setting range	DPB	23.95	mm Hg

CALCULATIONS			
	Symbol		Units
New EPA method (AP-42) *			
Breathing losses			
Tank vapor space volume	Vv	226.20	ft3
Vapor density	Wv	6.531E-03	lb/ft3
Vapor space expansion factor	KE	-0.01433	
Vented vapor saturation factor	Ks	0.9063	ft2
Breathing losses	LB	-	lb/yr
Working losses	Lw	145.58	lb/yr
Total losses	LT	**145.58**	lb/yr
Simplified method **			
Breathing losses			
Temperature expansion factor		#VALUE!	
Air displaced per day		#VALUE!	lbmole/day
Breathing losses	LB	#VALUE!	lb/yr
Working losses	Lw	#VALUE!	lb/yr
Total losses	LT	**#VALUE!**	lb/yr

* **New EPA method (Source AP-42 - Supplement E - October 1992)**

** **Simplified method (Adaptation of the new EPA method)**

Note - Cells in pink are input cells. All other cells are calculated cells.

Special Cases:

1. Insulated or underground Tanks: omit breathing losses (LB).
2. Heated Tanks: use actual liquid temp and range for ambient temp and range.
3. Indoor Tanks: use actual indoor temp and range; reduce solar insolation to 0.
4. Tanks with conservation vents: use AP-42 method and enter breather vent range.
5. Tanks with N2 pads: use AP-42 method and enter breather vent range.

Breather Vent Range = Pressure vent setting - vacuum vent setting

= 10"wc - (-0.5oz per sq in)

= (16)(.036)psi - (-.03psi) = .58+.03 psi = .61psi

Paint Solar Absorptance from AP-42

Paint Color	Paint Shade	Paint Factors	
		Paint Condition	
		Good	Poor
Aluminum	Specular	0.39	0.49
Aluminum	Diffuse	0.60	0.68
Gray	Light	0.54	0.63
Gray	Medium	0.68	0.74
Red	Primer	0.89	0.91
White	NA	0.17	0.34
Black	NA	1.00	1.00

TANK NO. V-3608

Table 1
TANK EMISSION CALCULATION FORM

Tank No.	**V-3608**	**Tank type**	**Horizontal fixed roof (insulat**	**Date**	**03/04/06**
Material stored	**Oxime**	**Company**	**DSM Chemicals**	**Performed by**	
City	**Augusta**	**State**	**GA**		
Description	**Outdoor storage tank**				

INPUT DATA	Symbol		Units
Vapor pressure Antoine constants			
Constant A		**9.0490**	
Constant B		**2,992.500**	
Constant C		**273.150**	
Molecular weight	Mv	**113.2**	Lb/lb-mole
Tank design data			
Shell height	Hs	**7.50**	ft
Diameter	D	**8.00**	ft
Liquid height		7.50	ft
Avg. Liquid height	HL	**3.75**	ft
Tank volume		2,820	gallons
Turnovers	N	**76**	
Net throughput	Q	215,712	gallons/yr
Tunover factor	KN	0.559	
Working loss product factor	Kp	**1.00**	
Meteorological data			
Daily ave. ambient temp.	TAA	**N/A**	°F
Daily max. ambient temp.	TAX	N/A	°F
Daily min. ambient temp.	TAN	N/A	°F
Daily ambient temp. range	DTA	**N/A**	°F
Tank paint solar absorptence	α	**N/A**	
Daily total insolation factor	I	**N/A**	Btu/ft2-day
Liquid bulk temperature	TB	240.00	°F
Daily vapor temp. range	DTv	10.00	°F
Daily ave. liquid surface temp.	TLA	240.00	°F
Daily max. liquid surface temp.	TLX	242.50	°F
Daily min. liquid surface temp.	TIN	237.50	°F
VP @ daily ave. liquid surf. temp.	PvA	22.4063	mm Hg
VP @ daily max. liquid surf. temp.	PvX	23.8661	mm Hg
VP @ daily min. liquid surf. temp.	PvN	21.0264	mm Hg
VP @ daily ave. ambient temp.	Pamb	N/A	mm Hg
Daily vapor pressure range	DPv	2.84	mm Hg
Breather vent pressure setting range		**0.46**	psia
Breather vent pressure setting range	DPB	23.95	mm Hg

CALCULATIONS	Symbol		Units
New EPA method (AP-42) *			
Breathing losses			
Tank vapor space volume	Vv	188.50	ft3
Vapor density	Wv	6.531E-03	lb/ft3
Vapor space expansion factor	KE	-0.01433	
Vented vapor saturation factor	Ks	0.9207	ft2
Breathing losses	LB	-	lb/yr
Working losses	Lw	140.81	lb/yr
Total losses	LT	**140.81**	lb/yr
Simplified method **			
Breathing losses			
Temperature expansion factor		#VALUE!	
Air displaced per day		#VALUE!	lbmole/day
Breathing losses	LB	#VALUE!	lb/yr
Working losses	Lw	#VALUE!	lb/yr
Total losses	LT	**#VALUE!**	lb/yr

* **New EPA method (Source AP-42 - Supplement E - October 1992)**

** **Simplified method (Adaptation of the new EPA method)**

Note - Cells in pink are input cells. All other cells are calculated cells.

Special Cases:

1. Insulated or underground Tanks: omit breathing losses (LB).
2. Heated Tanks: use actual liquid temp and range for ambient temp and range.
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5. Tanks with N2 pads: use AP-42 method and enter breather vent range.

Paint Solar Absorptance from AP-42

Paint Color	Paint Shade	Paint Factors	
		Paint Condition	
		Good	Poor
Aluminum	Specular	0.39	0.49
Aluminum	Diffuse	0.60	0.68
Gray	Light	0.54	0.63
Gray	Medium	0.68	0.74
Red	Primer	0.89	0.91
White	NA	0.17	0.34
Black	NA	1.00	1.00

Breather Vent Range = Pressure vent setting - vacuum vent setting

= 10"wc - (-0.5oz per sq in)

= (16)(.036)psi - (-.03psi) = .58+.03 psi = .61psi

ATTACHMENT 4

T-2801

Date	**Oxime ppm**
01/04/05	0.5
01/11/05	0.4
01/18/05	0.6
01/25/05	0.3
02/01/05	0.3
02/08/05	0.2
02/15/05	0.2
02/22/05	0.2
03/01/05	0.3
03/08/05	0.3
03/15/05	0.3
03/22/05	0.3
03/29/05	0.5
04/05/05	0.1
04/12/05	0.3
04/19/05	0.2
04/26/05	0.4
05/03/05	0.3
05/10/05	0.2
05/17/05	0.5
05/24/05	0.4
05/31/05	0.2
06/07/05	0.2
06/14/05	0.3
06/21/05	0.1
06/28/05	0.1
07/05/05	0.2
07/12/05	0.1
07/19/05	0.3
07/26/05	0.2
08/02/05	0.3
08/09/05	0.3
08/16/05	0.2
08/23/05	0.2
08/30/05	0.4
09/06/05	0.3
09/13/05	0.5
09/20/05	0.3
09/27/05	0.2
10/04/05	0.4
10/11/05	0.2
10/18/05	0.4
10/25/05	0.2
11/01/05	0.3
11/08/05	0.3
11/15/05	0.1
11/22/05	0.2
11/29/05	0.3
Average	**0.28**
MDL	**0.17**
	Oxime in lactam

Method
DCNA-10-CP009